

Data Validation Report

TDD No: 09-04-01-0011
 PAN: 001275.0440.01TA
 Site: El Dorado Hills
 Laboratory: Lab/Cor, Inc.
 Reviewer: Denise A. Shepperd, Trillium, Inc.
 Date: January 28, 2005

I. Case Summary

SAMPLE INFORMATION:

Asbestos Samples: LC-SRA-R01-1000404; LC-SRA-R02-100404; LC-SRA-R03-100404; LC-SRA-R04-100404; LC-SRA-R05-100404; LC-SRA-R103-100504; LC-SRA-R04-100504; LC-SRA-R03-100504; LC-SRA-R02-100504; LC-SRA-R01-100504; LC-SRA-R05-100504; LC-SRA-R01-100604; LC-SRA-R102-100604; LC-SRA-R03-100604; LC-SRA-R05-100604; LC-SRA-R04-100604; LC-SRA-R02-100604; LC-SRA-R04-100704; LC-SRA-R03-100704; LC-SRA-R05-100704; LC-SRA-R02-100704; LC-SRA-R01-100704; LC-SRA-R103-100804; LC-SRA-R04-100804; LC-SRA-R05-100804; LC-SRA-R02-100804; LC-SRA-R03-100804; LC-SRA-R01-100804; LC-NRA-R01-100904; LC-NRA-R02-100904; LC-NRA-R04-100904; LC-NRA-R05-100904; LC-NRA-R03-100904; LC-NRA-R101-101004; LC-NRA-R01-101004; LC-NRA-R05-101004; LC-NRA-R04-101004; LC-NRA-R03-101004; LC-NRA-R02-101004; LC-NRA-R03-101104; LC-NRA-R04-101104; LC-NRA-R05-101104; LC-NRA-R01-101104; LC-NRA-R02-101104

Matrix: 44 Air samples
 Analysis: Asbestos by Transmission Electron Microscopy
 Collection Dates: October 4 through 11, 2004
 Sample Receipt Date: October 15, 2004
 Analysis Date: November 24, 2004 through January 4, 2005
 Analytical Method: ISO Method 10312

FIELD QC:

Field Trip Blanks (TB): None
 Filter Blanks (FB): None
 Equipment Blanks (EB): None
 Background Samples (BG): None
 Field Duplicates (D1): Not Identified

TABLES:

1A: Analytical Results with Qualifications
 1B: Data Qualifier Definitions for Inorganic Data Review

SAMPLING ISSUES:

Three chain of custody (COC) documents were included in the data package and were properly completed. These documents included all of the field samples in the data package, as well as many additional samples.

VALIDATION PARAMETERS AND COMMENTS:

I. Holding Times, Preservation and Sample Integrity

This parameter is evaluated to ensure that sample custody is documented from collection through analysis, samples are analyzed within the recommended holding time, and that no alteration in sample content has occurred during sample shipment, handling, and storage.

There is no established holding time or storage condition for asbestos samples.

II. Calibration

The analyses of materials of known content ensures that identification and quantitation of analytes will be accurate for all samples. Review of the documentation provided for appropriate calibration determines whether or not the analytical results reported by the laboratory are valid and supported by the data.

The data deliverables for this project were included in multiple data packages. The calibration documentation was provided in a single separate package associated with all of the site sample data packages.

A letter representing documentation of an NVLAP laboratory site assessment conducted on 11/7/03 was included in the calibration data package. The letter included (dated 5/10/04) indicated that the laboratory met the on-site assessment requirements.

Results and evaluator notes and tables were included for an NISTIR 5351 analysis of an inter-laboratory QC sample. The laboratory's raw data were compiled and assessed by Batta Labs. Analysts were identified by initials and included all of the initials documented with this sample set, except "TM" and "JH." According to the assessor's notes, the sample included chrysotile fibers and structures and the laboratory's results were within NVLAP and NISTIR 5351 acceptance limits. No raw data were provided for this QC sample.

Results for a New York State Department of Health Environmental Laboratory Approval Program proficiency test, conducted between 9/7/04 and 11/9/04, were included. The proficiency samples included asbestos in air. The laboratory's results were satisfactory for all four of the air sample categories. Actinolite and amosite fiber types were identified and counts were acceptable according to the data sheet. No raw data were provided for this proficiency sample.

Acceptable instrument calibration was documented in the data package, including screen and camera magnification, camera length and camera constant, spot size, k-factor, beam dose, EDS sensitivity and peak intensity. No documentation of grid opening size was provided. Documentation was provided in the separate proficiency and calibration data package for October through December, 2004, for both of the instruments used for analysis of samples included in this data package. Analyses of the samples in this data set were performed during this time period.

Based on the fact that the laboratory demonstrated proficiency in the performance evaluation (PE) analyses performed in the third quarter of 2004, and that these PE samples included the two predominant asbestos types

detected in this field sample set, no action was taken by the validator. It is recommended however, that supporting data be expanded to include raw data supporting the identification of all asbestos types detected in PE samples and demonstration, wherever possible, of the correct identification (in known reference materials) of all fiber types detected in a field sample set.

III. Blanks

Sample matrices known to be devoid of the analytes of interest (method blanks) are prepared and analyzed with each analytical batch. Evaluation of this parameter ensures that contamination introduced during preparation and analyses is not attributed to the field samples.

Other blanks may be generated in the field or laboratory to ensure that no contamination is introduced during sampling and/or storage.

Blanks required for this project included Filter Blanks and Field Trip Blanks. No Filter Blanks or Field Trip Blanks were included with this sample set.

IV. Spiked Samples

The analytes of interest are added in known concentrations to like-matrix blanks or authentic field samples before preparation. This parameter is evaluated in order to assess the laboratory's ability to preserve and recover the compounds of interest.

The analytical method does not require laboratory spiked sample analyses. It is recommended by the validator that some type of laboratory prepared or purchased spiked analyses be performed with each analytical sample batch.

The project requirements specified that results from the most recent inter-laboratory study would be acceptable as an LCS sample for these data. This requirement was met by the laboratory and reported results for the inter-laboratory study sample were acceptable for all air sample parameters (see Section I).

V. Duplicate/Replicate Samples

Results for duplicate/replicate samples are evaluated to assess the laboratory's precision for the analytes of interest in the applicable sample matrix. For asbestos analyses, duplicate and replicate measurements take the form of a combination of variables which include the preparation of the grid, the choice of grid openings to be analyzed, and the analyst performing the counting and identification of structures.

The laboratory included all of the QC samples from all of the field sample sets in a separate data package under a separate report number.

The two analysts, JH and TM, not represented in the PE sample analyses included with the data packages for this project did perform intra-laboratory replicate and duplicate analyses on associated field samples. Results for these QC analyses for both analysts were within the sample-specific acceptance limits.

The quality assurance project plan (QAPP) requires five types of laboratory duplicate/replicate analyses, each to be performed at a rate of 5% (one for every twenty) of the field samples. Based on 44 field samples reported in the data package, two or more of each of these QC sample pairs were required. The laboratory compared the primary asbestos structure count for each of the QC samples prepared and analyzed. Results for all of the duplicate/replicate pair types were evaluated based on 95% confidence limits determined from the original sample count result. Results for all of the reported QC samples were within the laboratory's calculated limits. A summary of the laboratory QC samples included with this data set are as follows:

Replicate analyses:

- four samples, LC-SRA-R04-100504, LC-SRA-R102-100604, LC-SRA-R02-100804, and LC-NRA-R04-101104, were analyzed as replicates wherein a different preparation was analyzed by the same analyst;

Duplicate analyses:

- three samples, LC-SRA-R05-100604, LC-SRA-R02-100604, and LC-NRA-R02-101104, were analyzed as duplicates wherein the same grid openings were recounted by a different analyst;
- six samples, LC-SRA-R04-100404, LC-SRA-R103-100504 (analyzed twice as this type of duplicate, by a second and third analyst), LC-SRA-R02-100704, LC-NRA-R03-101104, and LC-NRA-R02-101104 were analyzed as duplicates wherein a different analyst counts different grids; and
- four samples, LC-SRA-R04-100504, LC-SRA-R102-100604, LC-SRA-R02-100804, and LC-NRA-R04-101104, were analyzed as duplicates wherein a new preparation was counted by a different analyst.

No samples were analyzed as QC samples for one of the required categories:

- a replicate wherein different grid openings were selected by the same analyst for a second measurement

Two or more QC samples should have been included for each of these five QC sample categories in order to satisfy the 5% requirements of the QAPP.

An additional type of QC sample, not identified by the QAPP, was included. Samples LC-SRA-R05-100604, LC-SRA-R02-100604, LC-NRA-R03-101104, and LC-NRA-R02-101104 were recounted by the same analyst, counting the same grids.

Only results for total count of primary structures were included on the QC summary table and the reporting forms in the QC package. The validator compared reported results for primary structure counts as well as the raw data count sheets for the original field samples and paired QC samples. Overall agreement between total counts and identification of asbestos types was good. There were some significant differences between morphological types identified and measured dimensions by both the same and a different analyst recounting the same grid openings. For instance, according to the raw data count sheets, the initial analyst counting sample LC-SRA-R02-100604 identified discrete fiber and 9 disperse matrices, for a total structure count of 10. The same analyst, recounting the same grids at a later date identified 14 discrete fibers and 1 disperse matrix, for a total count of 15. A second analyst recounting the same grid openings identified 5 discrete fibers and 10 disperse matrices, for a total count of 15. Similar results were seen for other QC sample sets associated with this project. Because these identifications are reflected in the counts for the various asbestos categories, the data user should keep this variability in mind if results for the individual categories are going to be used.

The QC summary form listed two different results (12 and 9 primary structures) for the initial sample analysis of LC-NRA-R02-101104. According to the report form and count sheet for this sample, 9 is the correct result. The QC summary was corrected to reflect the correct sample result and corrected acceptance limits based on that result.

The date of analysis listed on the report form for sample LC-SRA-R05-100604 in the QC data package was 11/4/04. Because this date is considerably earlier than any of the other analyses performed with this data set (analyses began 11/24/04) it is likely that this date is a misprint. At the data user's discretion the laboratory should be requested to provide the correct analysis date for this QC sample.

According to the QAPP provided with the data packages, field duplicates were required at a rate of 10% of field samples. Field duplicate pairs were not identified or evaluated as part of this validation effort.

VI. Identification

Identification of asbestos structures and fibers is dependent on sample preparation techniques, analyst training, instrument operation, and data interpretation. Comparison with results from known standards is used to evaluate the accuracy of the structure identification for field samples.

Chrysotile, actinolite, amosite, anthophyllite, winchite, and tremolite were reported by the laboratory in the field samples. According to the report forms provided in the separate QC package, the laboratory correctly identified actinolite, chrysotile, and amosite in PE sample analyses performed in the third quarter of 2004. Comparison of identification between the various analysts, grid opening, and preparations combinations that make up the daily QC for these analyses were acceptable. Therefore; based on the documentation provided, fiber and structure identifications for chrysotile, amosite, and actinolite were determined to be valid as reported. It was assumed that the laboratory correctly identified the other amphibole structures that were reported in the field samples.

For a discussion of the variation observed in the identification of asbestos structures and morphological types see the previous section (section V).

VII. Quantitation and Reported Detection Limits

Raw data documentation is reviewed to ensure that all reported results and detection limits are correctly calculated, accurately reported, and supported by the raw data. Results for asbestos categories, fiber density, and detection limits were correctly calculated and accurately reported by the laboratory. Results were verified by the validator using the information included on the reporting forms and the chain of custody records.

Results from the analysis of one of the field samples were not included in the data package. Sample LC-SRA-R01-100704 was rejected by the laboratory at preparation because the filter was blown out.

VIII. System Performance

This parameter is evaluated to ensure that the laboratory analytical systems were functioning properly at the time of analyses and that methodology appropriate to the analyses were followed.

The analytical systems appear to have been working satisfactorily and to have been calibrated properly at the time of these analyses, based on the available documentation.

IX. Documentation

Data and documentation completeness is critical in providing support for the reported results. Problems encountered with the nature or quality of the data package documentation are addressed.

No raw data were provided in the data package for the proficiency samples analyzed in support of the laboratory's accreditation. Raw data to support the identification of actinolite and amosite were received upon request on 1/26/05.

Raw data for chrysotile fibers were not included in the data package for review. Raw data documenting fiber identification for the other asbestos types identified in the field samples were present in the data package. Upon request representative raw data for chrysotile from each sample set were supplied by the laboratory on 1/27/05.

Some raw data for chrysotile fibers were not included in the data package for review. Provided were several EDS for chrysotile; worksheets on mineral composition, and the negatives from the TEM were not provided. Raw data

documenting fiber identification for the other asbestos types identified in the field samples were present in the data package. Upon request, EDS and negatives for chrysotile in selected samples from each sample set were supplied by the laboratory on 1/27/05.

Raw data for sample AAMS-D12-100804 in the form of TEM negatives and ED spectra were present in the data package. This sample was not included for analysis with this data set.

Some of the ED spectra copies were illegible due to apparent copying problems.

Count sheets included in the data package are computer generated forms. No date of the actual count is presented on these forms. If there is a corresponding bench sheet from which these forms are prepared, these should be supplied as a part of the data package. It is recommended that analyst's initials and date of count be added to the documentation.

The legend for the count sheets, which defines the codes used for the structure counts, lists PSCH as the code for protocol chrysotile structures. The code actually appearing on the count sheets for this category is PCAS.

Raw data are an integral part of a complete and defensible data package. Edits made on all data should be performed correctly. Proper editing requires drawing a single line through the incorrect information, adding the correct information, and initialing and dating the changes.

Asbestos structures identified in the field samples included actinolite, chrysotile, amosite, anthophyllite, winchite, and tremolite. Examples of known materials included in the data package in support of the sample analyses included only actinolite, chrysotile, and amosite, identified in the proficiency sample analyses. Based on the documentation provided, the identification of the other amphibole fiber types in a known standard was not documented.

COMMENTS:

- A. Two different results were listed on the QC summary sheet for the original analysis of sample LC-NRA-R02-101104. The result from the sample report form and count sheet is 9 primary structures; the QC summary lists both 9 and 12 primary structures counted.

ADDITIONAL COMMENTS:

Based on the available data, results for all of the samples included in this data set were determined to be valid as reported by the laboratory. Reported results, analytical sensitivity, and detection limits are considered to be accurate within the bounds of the 95% confidence limits determined for each sample.

The data results tables included as Table 1A include only the primary and total asbestos structure counts. Counts for individual categories required by the project Scope of Work are presented in the associated electronic data deliverables (EDD) tables.

This report was prepared according to the specifications of the analytical method, ISO Method 10312 "Ambient air - Determination of asbestos fibres - Direct-transfer transmission electron microscopy method," the document "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," 2/94, and Trillium, Inc.'s SOP No. 0497-06A, for Validation of Analytical Data: Inorganic Analytes.

TABLE 1B

DATA QUALIFIER DEFINITIONS FOR INORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared in accordance with the document, "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," 2/94.

- U The analyte was analyzed for, but was not detected above the level of the reported value. The reported value is either the sample quantitation limit or the sample detection limit.
- L Indicates results which fall between the sample detection limit and the CRDL. Results are estimated and are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of detection.
- J The associated value is an estimated quantity. The analyte was analyzed for and was positively identified, but the reported numerical value may not be consistent with the amount actually present in the environmental sample.
- R The data are unusable. The analyte was analyzed for, but the presence or absence of the analyte cannot be verified.
- UJ A combination of the "U" and "J" qualifier. The analyte was analyzed for but was not detected. The reported value is an estimate and may be inaccurate or imprecise.